International Standard 8008

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Aluminium oxide primarily used for the production of aluminium — Determination of specific surface area by nitrogen adsorption — Single-point method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Détermination de l'aire massique (surface spécifique) par adsorption d'azote — Méthode à un point de mesure

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Foreword

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Aluminium oxide primarily used for the production of aluminium — Determination of specific surface area by nitrogen adsorption — Single-point method

1 Scope and field of application

This International Standard specifies a single-point method for the determination, by nitrogen adsorbtion, of the specific surface area of aluminium oxide used for the production of metal.

The lower limit is 0,1 $m^2 \cdot g^{-1}$.

The upper limit of the present method may be 1 000 $m^2 \cdot g^{-1}$ or greater, but those values are not of interest to the aluminium industry.

The range of measurement depends on the dimensions of the apparatus.

2 References

ISO 803, *Aluminium oxide primarily used for the production of aluminium — Determination of aluminium at* 300 °C *(conventional moisture).*

ISO 2927, *Aluminium oxide primarily used for the production of aluminium — Sampling.*

3 Principle

The method is based on the property of the solids to adsorb gas molecules at their surface.

In the range between 0,05 and 0,3 times the saturated vapour pressure of the measuring gas, the multilayer adsorption begins. The formation of the first monolayer of adsorbed molecules can be detected from the behaviour of the adsorption isotherm in this range; this is the only process relevant to the present method.

Nitrogen is introduced at ambient temperature and at atmospheric pressure into two bottles of equal volume one of which contains the test portion while the other is empty. The connected bottles are immersed in refrigerant bath of liquid nitrogen. Since some nitrogen is adsorbed by the sample a differential pressure occurs between the two bottles and is measured by means of a differential manometer. From this difference, the number of molecules adsorbed on the surface is calculated.

This number is multiplied by the known area occupied by a single adsorbed molecule so that the total surface area is obtained. The area occupied by an absorbed nitrogen molecule is taken as $16{,}2 \times 10^{-20}$ m^2.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Adsorption apparatus (see figure 1).

The apparatus consists of a reference bottle (7) and a sample adsorption bottle (8) which are moved onto the two connecting pieces, with sealing rings in between to make the joint gas-tight. At each connecting piece, there is a valve (1 and 2), by which the bottles can be connected to the atmosphere. The measuring gas is admitted to the bottle through the capillaries inside the connecting pieces.

The bottles made of shock-resisting glass have a volume of about 100 cm^3. The difference between the volume of the two bottles shall not exceed 0,1 %. The bottles' necks consist of calibrated glass tubes with an internal diameter of 9 ± 0,02 mm. Each tube has an upper and lower mark.

In this way, several bottles can be used as either sample or reference bottles without compensating the volumes for each combination of bottles. A differential manometer containing dibutyl phthalate is arranged between the two bottles. The legs of the differential manometer are connected to the two inlet capillaries of the adsorption bottles. By means of the valve 4, the two bottles, i.e. the two inlet capillaries, can be separated from or connected to each other. By means of the valve 5, the liquid in the two legs of the differential manometer can be separated or connected. The legs of the differential manometer consist of calibrated glass tubes with an internal diameter of 5 ± 0,02 mm. This ensures that the change in volume during the adsorption measurement can be calculated with sufficient accuracy. The inlet capillary of the sample bottle, being the shorter of the two, is connected to a compensating volume (10), which is adjusted during preparation of the apparatus.

The measuring gas is admitted to the apparatus via the valve 3. If the valves 1, 2 and 4 are open, the measuring gas passes through both bottles. If the valves 1 and 4 are closed, the reference bottle is shut off and only the sample bottle is purged with the measuring gas.

During measurement, only a part of the gas volume, which is downstream of the valve 3 and upstream of the valves 1 and 2, is cooled by liquid nitrogen to the measuring temperature. The gas volume remaining at room temperature shall be limited to 10 % of the total volume at maximum. For this reason, the connections to the adsorption bottles are capillaries, which occupy most of the necks of the bottles in order to keep the portion remaining at room temperature as small as possible.